PCT

WORLD INTELLECTUAL PROPERTY ORGANIZATION International Bureau



INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(51) International Patent Classification 7:

(11) International Publication Number:

WO 00/38918

B32B 27/12, D04H 13/00

A1

(43) International Publication Date:

6 July 2000 (06.07.00)

(21) International Application Number:

PCT/US99/25972

(22) International Filing Date:

4 November 1999 (04.11.99)

(30) Priority Data:

09/221,079

US 28 December 1998 (28.12.98)

(71) Applicant (for all designated States except US): KIM-BERLY-CLARK WORLDWIDE, INC. [US/US]; P.O. Box 349, 401 North Lake Street, Neenah, WI 54956 (US).

(72) Inventors; and

- (75) Inventors/Applicants (for US only): TAYLOR, Jack, D. [US/US]; 135 Big Creek Court, Roswell, GA 30076 (US). HAFFNER, William, B. [US/US]; 1256 Tinderbox Lane, Kennesaw, GA 30144 (US).
- (74) Agent: BERNSTEIN, Jason, A.; Bernstein & Associates, P.C., 30 Perimeter Center East, Suite 121, Atlanta, GA 30346-1902 (US).

(81) Designated States: AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MD, MG, MK, MN, MW, MX, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, US, UZ, VN, YU, ZA, ZW, ARIPO patent (GH, GM, KE, LS, MW, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).

Published

With international search report.

- (54) Title: HEAT SHRINKABLE ELASTOMERIC LAMINATE MATERIAL
- (57) Abstract

A laminate having improved extensibility is made of an elastic sheet layer prestretched and bonded to at least one extensible layer. In a first embodiment, the elastic layer is bonded in selected locations to an extensible layer and then heated. The latent extensibility of the prestretched elastic layer and the extensibility of the extensible layer produce desired extensibility useful as waistbands, side panels and side tabs in diapers and training pants. Diapers and training pants made with such a material can fit a larger number of body sizes because of the improved extensibility. In a second embodiment the film is prestretched in the cross direction and the machine direction. then laminated to an extensible layer, resulting in two-way extensibility. The elastic layer can be apertured. Aperturing produces a laminate that is breathable, while maintaining improved extensibility.

FOR THE PURPOSES OF INFORMATION ONLY

Codes used to identify States party to the PCT on the front pages of pamphlets publishing international applications under the PCT.

SI SK SN SZ TD TG TJ TM TR TUA UC US VN YU ZW

Togo Tajikistan Turkmeni

Turkey Trinidad and Tobago Ukraine

Utraine
Uganda
United States of America
United States of America
United States
United States
United States
Viet Nam
Yugoslavia
Zimbabwe

AL	Albania	ZS	Spain	LS	Lesotho
AM	Armenia	PI	Pintand	' LT	Lithpania
AT	Austria	PR	Prance	w	Locambourg
ΑŲ	Australia	GA	Gabon	LV	Latvia
SA	Azerbaijan	CB	United Kingdom	MC	Moseco
BA	Bosnia and Herzegovina	CE	Georgia	MID	Republic of Moldova
BB	Bertrados	CH	Chena	MC	Madagascar
34	Belgium	GN	Cuinea	MIK	The former Yugoslav
BF	Burkina Faso	GR	Grocce		Republic of Macedonia
BG	Bulgaria	HU	Hungary	ML	Meli
BJ	Benin	(E	Ireland	MN	Mongolia
BR	Brazil	IL	Israel .	· MR	Mauritania
BY	Belarus	15	Iceland	MW	Malawi
CA	Canada	IT	Italy	MX	Mexico
CF	Central African Republic	jp	Japan,	NB	Niger
CC	Congo	KE	Konya	NL	Netherlands
CH	Switterland	KG	Kyrgyzstan	NO	Norway
CI	Citte d'Ivoire	KP	Democratic People's	NZ	New Zealand
CM	Cameroon		Republic of Korea	' PL	Poland
CN	China	KR	Republic of Korea	PT	Porregal
cu	Cuba	KZ	Kazakstan	RO	Romania
CZ	Czech Republic	ıc	Saint Lucia	RU	Russian Federation
DΕ	Germany	u	Liechtenstein	SD	Sudan
DK	Denmark	LK	Sri Lanka	SE	Sweden
23	Estonia	LR	Liberia	SG	Singapore

WO 00/38918

PCT/US99/25972

5 .

HEAT SHRINKABLE ELASTOMERIC LAMINATE MATERIAL

10

15

FIELD OF THE INVENTION

The present invention relates to nonwoven laminate materials having improved extensibility and recovery, while maintaining strength. More particularly, the present invention provides a laminate of an elastic sheet and an extensible sheet. The laminate is heated to induce shrinking and optionally can be apertured.

BACKGROUND OF THE INVENTION

It is a goal of diaper makers to have one size diaper fit the largest number of users, so as to reduce the number of different diapers needed, which in turn reduces shelf space and inventory needs in stores. Addressing this need has resulted in the side pieces (also known as the ears or tabs) of diapers being designed with a maximum amount of stretch, while retaining strength and tear resistance properties. New materials and treatment methods have been developed to address this need. Often, the consequence of improving elasticity is reduced strength as the compromise

in material characteristics. There is a continuing need for improved elasticity in the tabs while maintaining strength.

Elastic materials are desired for personal care and other garment-like products to provide desirable conformance to the wearer. Two processes that readily produce low cost elastic sheet materials are film extrusion and meltblowing.

Normal nonwoven fabrics such as normal spunbond fabrics are not particularly elastic. Laminates of normal spunbond fabrics and elastic films or elastic meltblown fabrics possess negligible elasticity. Extensible fabrics such as extensible nonwoven fabrics, e.g., extensible necked, spunbond fabrics are extensible in the direction of their extensibility, but they exhibit negligible recovery. A laminate of an extensible fabric and an elastic film or elastic meltblown fabric does have extensibility as well as recovery. However, there are inherent limits on the maximum extension and recovery that can be obtained with extensible fabrics and elastic films or elastic meltblown fabrics. It would be desirable to use laminates of extensible materials and elastic films or elastic meltblown fabrics and impart greater extensibility and recovery properties.

15

20

Some elastic film materials have a certain amount of "latent" extensibility inherent therein. To "activate" this extensibility the film is often heated, causing it to shrink. When pulled, the activated film does extend and recover. Forming a laminate of such an elastic film and a non-extensible fabric results in a material that has limited extensibility. However, heating this material causes it to shrink, resulting in a fabric which can stretch to its original, unheated size. Even with this stretch, there is still a need for improvement. It would be desirable to improve the stretch characteristics of such elastic film to non-extensible fabric laminates while not substantially changing material composition or overall production technology.

WO 00/38918 SUMMARY OF THE INVENTION

10

Generally described, the present invention provides in a first embodiment an elastic sheet layer that is provided as being pre-stretched in the cross direction (CD). At least one CD extensible layer and the elastic sheet are laminated to each other. The multilayer material is then preferably heated, which shrinks the elastic sheet in the CD direction and causes CD gathering of the CD extensible layer. This material has superior CD direction extensibility based on both the latent energy in the film and the extensibility in the extensible layer.



In a second embodiment, the prestretched film is prestretched and set in both the CD direction and the machine direction ("MD"). At least one extensible layer, which is extensible in either the CD direction or the MD direction or both the CD and MD directions, and the elastic sheet are laminated to each other as done in the first embodiment. The laminate is heated to induce film layer shrinkage in both the CD and MD directions, and gathering of the extensible layer. The material produced in this manner will have extensibility in both the CD and MD directions. The material has superior extensibility in the direction or directions that the extensible layer is extensible, based on both the latent energy in the film and the extensibility in the extensible layer.

In a third embodiment, an elastic sheet layer is provided as being pre-stretched in the MD direction only. At least one MD extensible layer and the elastic sheet are laminated to each other. The multilayer material is then preferably heated, which shrinks the elastic sheet in the MD direction and causes MD gathering of the MD extensible layer. This material has superior MD direction extensibility based on both the latent energy in the film and in the extensible layer.

In a fourth embodiment, the elastic sheet layer, e.g., elastic film, in the multilayer material is apertured. This aperturing produces a multilayer material that is breathable, while



maintaining improved extensibility.

Accordingly, it is an object of the present invention to provide a fabric which has improved extensibility and retractability.

It is another object of the present invention to provide a material suitable for use as side panels in diapers, training pants, and incontinent protection products, and the like.

It is a further object of the present invention to provide a heat shrinkable laminate material comprising a heat shrinkable elastic film and at least one extensible layer.

It is another object of the present invention to provide a material suitable for use as tabs in diapers or similar products.

It is a further object of the present invention to provide a fabric that has desirable extensibility, and can function as a liner, yet permit passage of fluids and semi-fluids, such as urine and feces.

Other objects, features, and advantages of the present invention will become apparent upon reading the following detailed description of embodiments of the invention, when taken in conjunction with the appended claims.

15 DESCRIPTION OF THE INVENTION

DEFINITIONS

As used herein the term "nonwoven" fabric or web means a web having a structure of individual fibers or threads which are interlaid, but not in a regularly repeating pattern as in a meshed or knitted fabric. Nonwoven fabrics or webs have been formed by many processes such as, for example, meltblowing processes, spunbond processes, hydroentangling, air-laid and bonded carded web processes.

4

As used herein the term "spunbond fibers" refers to small diameter fibers which are formed by extruding molten thermoplastic material as filaments from a plurality of fine, usually circular capillaries of a spinneret with the diameter of the extruded filaments then being rapidly reduced as described, for example, in U.S. Patent 4,340,563 to Appel et al., and U.S. Patent 3,692,618 to Dorschner et al., U.S. Patent 3,802,817 to Matsuki et al., U.S. Patents 3,338,992 and 3,341,394 to Kinney, U.S. Patent 3,502,763 to Hartman, U.S. Patent 3,542,615 to Dobo et al, and U.S. Patent No. 5,382,400 to Pike et al. Spunbond fibers are generally not tacky when they are deposited onto a collecting surface and are generally continuous. Spunbond fibers are often about 10 microns or greater in diameter. However, microfiber spunbond may be achieved by various methods including, but not limited to, those described in commonly assigned U.S. Patent Application No. 08/756,426 filed November 26, 1996, continuation patent application filed April 30, 1998, by Mormon et al. and U.S. Patent No. 5,759,926 to Pike et al.

As used herein the term "meltblown fibers" means fibers of polymeric material which are generally formed by extruding a molten thermoplastic material through a plurality of fine, usually circular, die capillaries as molten threads or filaments into converging high velocity, usually hot, gas (e.g., air) streams which attenuate the filaments of molten thermoplastic material to reduce their diameter. Thereafter, the meltblown fibers can be carried by the high velocity gas stream and are deposited on a collecting surface to form a web of randomly dispersed meltblown fibers. Such a process is disclosed, for example, in U.S. Patent 3,849,241 to Butin et al. Meltblown fibers may be continuous or discontinuous and are generally tacky when deposited onto a collecting surface. Meltblown fibers can include microfiber webs.

15

As used herein the term "polymer" generally includes but is not limited to, homopolymers, copolymers, such as for example, block, graft, random and alternating copolymers, terpolymers, etc., and blends and modifications thereof. Furthermore, unless otherwise specifically limited,

the term "polymer" includes all possible spatial configurations of the molecule. These configurations include, but are not limited to isotactic, syndiotactic and random symmetries.

As used herein, the term "machine direction" or MD means the length of a fabric in the direction in which it is produced. The term "cross machine direction" or CD means the width of fabric, i.e., a direction generally perpendicular to the MD.

As used herein, the term "transverse direction" means transverse to the direction in which the material is stretched.

As used herein the term "extensible" means elongatable or stretchable in at least one direction.

As used herein the term "recover" refers to a contraction of a stretched material upon termination of a biasing force following stretching of the material by application of the biasing force. For example, if a material having a relaxed, unbiased length of 1 inch (2.54 cm) was elongated 50 percent by stretching to a length of 1.5 inches (3.81 cm), the material would have a stretched length that is 150 percent of its relaxed length. If this exemplary stretched material contracted, that is recovered to a length of 1.1 inches (2.79 cm) after release of the biasing and stretching force, the material would have recovered 80 percent (0.4 inch (1.02 cm)) of its elongation.

10

As used herein "elastic" means a material which, upon application of a biasing force, is stretchable, that is extensible, to a stretched, biased length which is at least 150% of its relaxed unbiased length, and which will retract at least 50 percent of its elongation upon release of the elongating force. A hypothetical example would be a 1 inch (2.54 cm) sample of a material which is elongatable to at least 1.50 inches (3.81 cm) and which, upon release of the biasing force, will retract to a length of not more than 1.25 inches (3.18 cm).

6

As used herein, the term "inelastic" or "nonelastic" refers to any material which does not fall within the definition of "elastic" above.

As used herein, the term "neckable material" refers to any material which can be necked.

As used herein, the term "necked material" refers to any material which has been constricted in at least one dimension by processes such as, for example, drawing or gathering.

As used herein, the term "necking" refers to any of several processes known to those skilled in the art, for example, passing one end of the material through a first niproll assembly and then through a second niproll assembly, wherein the speed of the second niproll assembly is faster than the speed of the first niproll assembly. The speed differential causes the material to stretch between the niproll assemblies in the machine direction, thereby causing the material to neck in.

As used herein, the term "personal care product" means diapers, training pants, absorbent underpants, adult incontinence products, feminine hygiene products and like performing products.

DETAILED DESCRIPTION OF THE PREFERRED EMBODIMENTS

In general, the present invention provides an elastic, fibrous nonwoven laminate comprising an elastic film layer and at least a first extensible fibrous nonwoven web facing layer. If desired, additional extensible layers may be attached to the laminate as, for example, a second extensible fibrous nonwoven facing layer on a surface of the elastic substrate layer which is opposed to the first extensible facing layer. For purposes of clarity the term "layer" will generally refer to a single piece or sheet of material, but the same term should also be construed to mean multiple sheets or plies of material which, together, form one or more of the "layers" described herein.

The extensible nonwoven layer is preferably formed of a spunbond. The spunbond material

is comprised of a polyolefin selected from the group consisting of polypropylenes, polyethylenes, and copolymers of propylene and ethylene suitable for spunbond processing. A preferred polypropylene is available as Exxon® PD 3445 polypropylene (hereinafter sometimes referred to as "PP"), available from Exxon Chemical Company, Houston, Texas. It was also found that blending the Exxon® PD 3445 with a lower viscosity polypropylene typically used for meltblowing applications, such as Montell® PF 015 polypropylene (hereinafter sometimes referred to as "Montell® PD 015"), available from Montell Chemical, Wilmington, Delaware, where the Exxon® PD 3445 was present in a range of approximately 50-100%, more preferably approximately 66%, provided an acceptable mix. It was found that 100% Exxon® PD 3445 provided a higher quality result than using a blend of polypropylene resins of narrow molecular weight distributions with lower melt viscosities, e.g., MF (at 230°C) is greater than about 35 grams/10 minutes. It is to be understood, however, that for certain purposes such a blend can be employed. Where a copolymer of propylene and ethylene is used, the ethylene content is present in a concentration of approximately 7% or less with approximately 93% or more of propylene. It is to be understood that bicomponent, multicomponent, and multiconstituent fibers can be used with the present invention.

10

15

20

The spunbond fibers are produced according to one of the processes described hereinabove.

The elastic sheet is preferably made of an elastomeric polymer selected from the group consisting of elastic polyesters, elastic polyurethanes, elastic polyamides, elastic polyolefins, metallocene catalyzed polyolefins, and elastic A-B-A' block copolymers, where A and A' are the same or different thermoplastic polymers, and where B is an elastomeric polymer block. A preferred polymer is KRATON® G-2755 compound, which is a blend of an elastomeric polymer, a polyolefin, and a tackifying resin. Any tackifier resin can be used which is compatible with the elastomeric polymer and can withstand the high processing (e.g.,

extrusion) temperatures. If blending materials such as, for example, polyolefins or extending oils are used, the tackifier resin should also be compatible with those blending materials. For the KRATON®-type elastomers, REGALREZTM and ARKONTM P series tackifiers are examples of hydrogenated hydrocarbon resins. ZONATAKTM 501 Lite is an example of a terpene hydrocarbon. REGALREZTM hydrocarbon resins are available from Hercules Incorporated. ARKONTM P series resins are available from Arakawa Chemical (U.S.A.) Incorporated. Of course, the present invention is not limited to use of such three tackifying resins, and other tackifying resins which are compatible with the other components of the composition and can withstand the high processing temperatures, can also be used.

A polyolefin may also be blended with the elastomeric polymer to improve the processability of the composition. The polyolefin must be one which, when so blended and subjected to an appropriate combination of elevated pressure and elevated temperature conditions, is extrudable, in blended form, with the elastomeric polymer. Useful blending polyolefin materials include, for example, polyethylene, polypropylene and polybutene, including ethylene copolymers, propylene copolymers and butene copolymers. A particularly useful polyethylene may be obtained from Equistar Chemicals of Cincinnati, Ohio under the trade designation PETROTHENE® NA601 (also referred to herein as PE NA601 or polyethylene NA601). Two or more of the polyolefins may be utilized.

10

20

A pressure sensitive elastomer adhesive formulation may include, for example, from about 20 to about 99 percent by weight elastomeric polymer, from about 5 to about 40 percent polyolefin and from about 5 to about 40 percent resin tackifier. For example, the composition KRATON® G-2755 includes, by weight, about 61 to about 65 percent KRATON® G-1659, about 17 to about 23 percent polyethylene NA-601 (available from Equistar Chemicals, Cincinnati, OH), and about 15 to about 20 percent REGALREZ® 1126. Other beneficial additives

may be flow modifiers to encourage the polymer melt to penetrate into the fiber structure, and surface tension modifiers to reduce elastomer melt and fiber structure surface tension differences. Flow modifiers may be added to the elastomeric polymer to reduce elastomer melt viscosity. Such a flow modifier for KRATON®-type elastomers are low molecular weight polyolefins.

The elastic sheet may be formed from, for example, (polystyrene/poly(ethylene-butylene)/polystyrene) block copolymers available from the Shell Chemical Company under the trademark KRATON® G. One such block copolymer may be, for example, KRATON® G-1659. Other exemplary elastomeric materials which may be used to form the elastic sheets include polyurethane elastomeric materials such as, for example, those available under the trademark ESTANE® from B. F. Goodrich & Co., elastomeric materials such as, for example, polyether-block-amides available under the trademark PEBAX® from Atochem Inc. Polymers Division (RILSAN®) (Glen Rock, New Jersey), and polyester elastomeric materials such as, for example, those available under the trade designation HYTREL® from E. I. du Pont De Nemours & Company. Formation of elastic sheets from polyester elastic materials is disclosed in, for example, U.S. Patent No. 4,741,949 to Morman et al. and U.S. Patent No. 4,707,398 to Boggs. Sheets made from thermoplastic elastomer compositions disclosed in U.S. Patent No. 5,182,069, commercially available as EXX-601 from Exxon Chemical Company, are also usable with the present invention. This material is provided as prestretched in the cross direction.

10

Films are discussed herein as a preferred material; however, heat shrinkable elastic foams, fibrous webs (e.g., meltblown), and heat-shrinkable woven or knitted elastic sheets, i.e., any heat shrinkable elastic sheet, can also be used and is contemplated as being within the scope of the present invention.

Elastomeric thermoplastic polymers useful in the practice of this invention may be those

made from block copolymers such as polyurethanes, copolyether esters, polyamide polyether block copolymers, ethylene vinyl acetates (EVA), block copolymers having the general formula A-B-A' or A-B like copoly(styrene/ethylene-butylene), styrene-poly(ethylene-propylene)-styrene, styrene-poly(ethylene-butylene)-styrene, poly(styrene/ethylene-butylene)-styrene, poly(styrene/ethylene-butylene) and the like.

5

Useful elastomeric resins include block copolymers having the general formula A-B-A' or A-B, where A and A' are each a thermoplastic polymer endblock which contains a styrenic moiety such as a poly (vinyl arene) and where B is an elastomeric polymer midblock such as a conjugated diene or a lower alkene polymer. Block copolymers of the A-B-A' type can have different or the same thermoplastic block polymers for the A and A' blocks, and the present block copolymers are intended to embrace linear, branched and radial block copolymers. In this regard, the radial block copolymers may be designated (A-B)_m-X, wherein X is a polyfunctional atom or molecule and in which each (A-B)_m-radiates from X in a way that A is an endblock. In the radial block copolymer, X may be an organic or inorganic polyfunctional atom or molecule and m is an integer having the same value as the functional group originally present in X. It is usually at least 3, and is frequently 4 or 5, but not limited thereto. Thus, in the present invention, the expression "block copolymer," and "A-B-A" and "A-B" block copolymer, is intended to embrace all block copolymers having such rubbery blocks and thermoplastic blocks as discussed above, which can be extruded (e.g., by meltblowing), and without limitation as to the number of blocks. The elastomeric nonwoven web may be formed from, for example, elastomeric (polystyrene/poly(ethylene-butylene)/ polystyrene) block copolymers. Commercial examples of such elastomeric copolymers are, for example, those known as KRATON® materials which are available from Shell Chemical Company of Houston, Texas. KRATON® block copolymers are available in several different formulations, a number of which are identified in U.S. Patents

4,663,220 and 5,304,599.

Polymers composed of an elastomeric A-B-A-B tetrablock copolymer may also be used in the practice of this invention. Such polymers are discussed in U.S. Patent 5,332,613 to Taylor et al. In such polymers, A is a thermoplastic polymer block and B is an isoprene monomer unit hydrogenated to substantially a poly(ethylene-propylene) monomer unit. An example of such a tetrablock copolymer is a styrene-poly(ethylene-propylene)-styrene-poly(ethylene-propylene) or SEPSEP elastomeric block copolymer available from the Shell Chemical Company of Houston, Texas under the trade designation KRATON® G-1730.

Another suitable material is a polyester block amide copolymer having the formula:

where n is a positive integer, PA represents a polyamide polymer segment and PE represents a polyether polymer segment. In particular, the polyether block amide copolymer has a melting point of from about 150°C to about 170°C, as measured in accordance with ASTM D-789; a melt index of from about 6 grams per 10 minutes to about 25 grams per 10 minutes, as measured in accordance with ASTM D-1238, condition Q (235°C/1Kg load); a modulus of elasticity in flexure of from about 20 Mpa to about 200 Mpa, as measured in accordance with ASTM D-790; a tensile strength at break of from about 29 Mpa to about 33 Mpa as measured in accordance with ASTM D-638 and an ultimate elongation at break of from about 500 percent to about 700 percent as measured by ASTM D-638. A particular embodiment of the polyether block amide copolymer has a melting point of about 152°C as measured in accordance with ASTM D-789; a melt index of about 7 grams per 10 minutes, as measured in accordance with ASTM D-1238, condition Q (235°C/1Kg load); a modulus of clasticity in flexure of about 29.50 Mpa, as

measured in accordance with ASTM D-639; and an elongation at break of about 29 Mpa, as measured in accordance with ASTM D-639; and an elongation at break of about 650 percent as measured in accordance with ASTM D-638. Such materials are available in various grades under the trade designation PEBAX®. Examples of the use of such polymers may be found in U.S. Patents 4,724,184, 4,820,572 and 4,923,742, to Killian et al. and assigned to the same assignee as the present invention.

Elastomeric polymers also include copolymers of ethylene and at least one vinyl monomer such as, for example, vinyl acetates, unsaturated aliphatic monocarboxylic acids, and esters of such monocarboxylic acids. The elastomeric copolymers and formation of elastomeric nonwoven webs from those elastomeric copolymers are disclosed in, for example, U.S. Patent No. 4,803,117.

The thermoplastic copolyester elastomers include copolyetheresters having the general formula:

15

20

where "G" is selected from the group consisting of poly(oxyethylene)-alpha,omega-diol, poly(oxypropylene)-alpha,omega-diol, poly(oxytetramethylene)-alpha,omega-diol and "a" and "b" are positive integers including 2, 4 and 6, "m" and "n" are positive integers including 1-20. Such materials generally have an elongation at break of from about 600 percent to 750 percent when measured in accordance with ASTM D-638 and a melt point of from about 350°F to about 400 °F (176 to 205°C) when measured in accordance with ASTM D-2117.

Commercial examples of such copolyester materials are, for example, those known as ARNITEL®, formerly available from Akzo Plastics of Arnhem, Holland and now available

from DSM of Sittard, Holland, or those known as HYTREL® which are available from E.I. du Pont de Nemours of Wilmington, Delaware.

The elastic sheet can be formed by any of several processes known to those skilled in the art, such as, but not limited to, extrusion. The elastic sheet can be formed prior to lamination and unrolled from a supply roll, or formed just prior (i.e., in the same processing step) to lamination. In a first embodiment, the elastic sheet is provided prestretched and set in the CD direction. The preferred material, EXX-601, is provided as a roll of prestretched material.

Athrodat onto

boing

15

20

The elastic sheet and the extensible layer, specifically the necked nonwoven layer are bonded together at spaced-apart locations using any of a number of bonding techniques known to those skilled in the art, such as, but not limited to, calendering, adhesives (blended in the components, on the surface of the components, between the components, dispersed in the matrix of the respective sheets, and the like), sonic and ultrasonic bonding techniques, hydroentangling, stitch bonding, through-air bonding, and the like.

The laminate is then heated to cause shrinking of the elastic sheet, which gathers the extensible nonwoven layer. Heating can be done at a temperature range of from about 49°C to about 68°C, more preferably from about 54°C to about 66°C, and still more preferably from about 60°C to about 66°C, for a period ranging from about 1 to about 6 minutes, more preferably, from about 1 to about 4 minutes, and still more preferably from about 1 to about 3 minutes. In a preferred embodiment, the laminate is heated at 66°C for 1-2 minutes. After cooling, the material is wound on a takeup roll. Alternatively, further processing or manufacturing steps can be taken at this time. In a production process, the heating step can be done after the laminate is cut and attached to the diaper or other end product, whereby the entire diaper is subjected to heat, such as by being placed in an oven.

It is also possible for the first layer to be a laminate of a thermoplastic elastomer capable of being made latently shrinkable, and an adhesive. The adhesive can be deposited onto the elastomer, coextruded with the elastomer, or laminated by other processes known to those skilled in the art.

The material of this first embodiment has stretch that is equal to the sum of the extension in the necked-in facings plus the stretchability achieved in the laminate as a result of the heat-shrinking of the elastic sheet. Thus, the amount of actual stretchability of the laminate material of the present invention can be much higher than that in laminates with non-extensible facings bonded to heat-shrinkable elastic sheeting, or with extensible facings, e.g., necked-in facings bonded to non-heat-shrinkable elastic sheeting. This is especially significant as many end-uses, such as stretchable waistbands and side panels in training pants, require materials with CD stretch much higher than can be achieved with the prior art, e.g., with just the extensibility that is present in necked-in nonwoven materials.

In a second embodiment the process of the first embodiment is utilized, however, the elastic sheet is prestretched and set in both the CD and MD directions. This imparts two-way extensibility in the CD and MD directions to the laminate material. The laminate is heated as discussed previously.

In a third embodiment, an elastic sheet layer is provided as being pre-stretched in the MD direction only. At least one MD extensible layer and the elastic sheet are laminated to each other. The multilayer material is then preferably heated, which shrinks the elastic sheet in the MD direction and causes MD gathering of the MD extensible layer. This material has superior MD direction extensibility based on both the latent energy in the film and in the extensible layer.

In a fourth embodiment of the present invention, the elastic sheet layer, e.g., elastic

film, in the multilayer material is apertured. Aperturing of the elastic film is achieved during the bonding step, e.g., during stitch-bonding of ultrasonic bonding when holes are punched through the elastic film at spaced apart locations and the facings are bonded to each other through the holes punched in the film. Other aperturing techniques include hydraulic needling, pin aperturing and the like, as are known to those of ordinary skill in the art. Multilayer material produced according to this fourth embodiment has desirable breathability and desirable water vapor transmission properties while maintaining improved extensibility. Multilayer material so produced can be used in personal care products, e.g., as waistbands and side panels in training pants and incontinence protection products.

It is to be understood that the various embodiments of the present invention, while discussing a single extensible layer being laminated to the prestretched and set elastic sheet layer, are contemplated as being able to incorporate a prestretched and set elastic sheet layer being laminated between two extensible layers, and also contemplates one or more layers of elastic sheets. In such an embodiment, a multilayer "sandwich" of elastic sheet layers and extensible layers are laminated to each other.

The invention will be further described in connection with the following examples, which are set forth for purposes of illustration only. Parts and percentages appearing in such examples are by weight unless otherwise stipulated.

EXAMPLES

Test Procedures

Tensile and Stretch Test

This method for tensile strength and stretch measures the breaking load and percent stretch at break of the laminate and detects the loads at various strain points on the curve. This is accomplished when the laminate is subjected to a continually increasing load in a single direction at a constant rate of speed. The test is done on a Sintech 2/S tester available from Sintech Corporation, 1001 Sheldon Dr., Cary, NC 27513. The test guidelines are as follows:

- Specimen size is 1 inch (2.54 cm) width by 6 inches (15.24 cm) length, the latter dimension
 is in the direction to be tested for tensile strength and stretch.
 - 2. Each specimen is placed lengthwise in jaw faces having a width of 3 inches(7.62 cm) and a height of 1 inch (2.54 cm), with a jaw span or separation of 3 inches (7.62 cm).
 - , 3. The crosshead speed is set at 12.0 inches (30.48 cm) per minute.
 - 4. Tensile strength is reported as grams per inch (2.54 cm) of specimen width.
- 15 5. Stretch is reported in percent.
 - 6. Break tensile strength percent drop is 1%.
 - 7. Break drop elongation is 0.02 inches (0.05 cm). (Break drop elongation is defined as that point where the tension has to drop 1% while elongation is over no more than a 0.02 inch (0.05 cm) run).

Water Vapor Transmission Rate ("WVTR")

5

10

20

The WVTR for the sample materials was calculated in accordance with ASTM Standard E96-80. Circular samples measuring three inches (7.62 cm) in diameter were cut from each of the test materials and a control which was a piece of Celgard® 2500 film from Hoechst Celanese Corporation of Sommerville, New Jersey. Celgard® 2500 is a microporous polypropylene film. Three samples were prepared for each material. The test cups were number 68-1 Vapometer cups distributed by Thwing-Albert Instrument Company of Philadelphia, Pennsylvania. One hundred milliliters of water were poured into each Vapometer cup and individual samples of the test materials and control material were placed across the open tops of the individual pans. The Vapometer cups were mechanically sealed along the edges of the cup, leaving the associated test material or control material exposed to the ambient atmosphere over a 6.8 cm diameter circle having an exposed area of approximately 33.17 cm². The cups were placed in a convection type oven at 100°F (37.7°C) for 24 hours. The oven was a constant temperature oven with external air circulating through it to prevent water vapor accumulation inside. After 24 hours, the pans were removed from the oven and weighed again. The preliminary test WVTR values were calculated with Equation (1) below:

(1) Test WVTR = (grams weight loss over 24 hours) x 315.5 g/m²/24 hours

The relative humidity within the oven was not specifically controlled. Under the predetermined set conditions of 100°F (37.7°C) and ambient relative humidity, the WVTR for the Celgard® 2500 control has been defined to be 5000 gsm for 24 hours. Accordingly, the control sample was run with each test and the preliminary test values were corrected to set conditions using Equation (2) below:

(2) Standardized WVTR = (Test WVTR/control WVTR) x (5000 $g/m^2/24$ hours).

18

Example 1: Comparative Example

Lamination of film to normal spunbond:

Lamination—Exxon® film EXX-601 (27 in. (68.6 cm) wide, 2 mil thickness (1 mil = 0.00254 cm) was laminated to normal (non-extensible), 0.4 osy (13.6 gsm) polypropylene spunbond fabric 29 inches (73.7 cm) wide, such that the film was ultrasonically bonded between two layers of the spunbond fabric by ultrasonically punching holes through the film and ultrasonically bonding the facings to each other. The film width at laminate windup was 26-26.25 inches (66.04-66.68 cm) (compared to 27 inches (66.58 cm) on film unwind roll). Line speed was 40 feet per minute (fpm) (1,219 cm/min). The material was smooth on the windup roll.

10 TESTING BY HAND

15

20

Heat shrinking—Three specimens, 1.5 inches (3.8 cm) wide in the MD direction and 140 mm long in the CD were cut out from a larger piece of the laminate. Two marks, 100 mm apart in the CD, were placed on all samples. The samples were then heated in a hot-air circulating oven at 66.6°C for 1.5 min. The distance between the two marks on each specimen was then measured and found to be, respectively, 55.0 mm, 55.0 mm, and 56.0 mm, with an average of 55.3 mm. The samples were then stretched by hand until the tension in the samples indicated that the specimens were fully extended, and the distance between the marks was again measured and found to be, respectively, 132.0 mm, 133.0 mm, 130.0 mm, with an average of 131.7 mm. The samples were allowed to relax for 2 min., and then again the distance between the respective marks was measured and found to be, respectively, 74.0 mm, 72.0 mm, and 70.0 mm, with an average of 72.0 mm.

The theoretical stretch was 100/55.3=1.81 or 81%. The actual stretch measured by hand was

131.7/55.3=2.38 or 138%.

TESTING BY MACHINE

A sample of the laminate measuring 18 inches (45.7 cm) in MD and 22 inches (55.9 cm) in CD was heat activated at 66°C for 3 minutes. After heat activation the sample measured 17 inches (43.2 cm) in the MD and 13.5 inches (34.3 cm) in the CD. The theoretical stretch in the heat activated laminate in the CD was 22/13.5= 1.63 or 63%.

Five specimens were cut from the heat activated laminate. Each specimen was cut to 1 inch (2.54 cm) width in MD by 6 inch (15.3 cm) length in CD, the latter length being in the direction of stretch measurement. Each specimen was tested by the Tensile and Stretch Test described hereinabove. The tensile strength in grams and percent stretch were measured for each specimen at various strain points on the curve, and the breaking tensile (break percent drop = 1%) and percent stretch at break (break drop elongation = 0.02 in. (0.05 cm)) were measured for each specimen. The averages of the tensile strength and percent stretch for the five specimens are shown in Table 1 below. The average percent stretch at break was 121%.

15

20

TABLE 1

							Break
% Stretch	0	25	50	75	100	119	121
Tensile Strength	0	481	705	1165	1699	1917	1909
as (grams/2.54 cm)							

Example 2-Lamination of film to necked spunbond:

Lamination—Exxon® film EXX-601 (27 inches (68.6 cm) wide, 2 mil thickness) was laminated to extensible, necked, 0.65 osy (22.04 gsm) polypropylene spunbond fabric 28 inches (71.1 cm) wide, such that the film was ultrasonically bonded between two layers of the spunbond

fabric by ultrasonically punching holes through the film and ultrasonically bonding the facings to each other. The film width at laminate windup was 27 inches (68.6 cm). The pattern roll was at ambient temperature (about 70°F (21.1°C)). Line speed was 40 feet per minute (fpm) (1219 cm/min). The material was smooth on the windup roll. The standardized WVTR of material on the windup roll was 938. Heat shrunk material with two facings had a usable basis weight of about 0.4 osy (13.56 gsm) to about 8 osy (271.2 gsm). The laminate of Example 2 had a basis weight of 4.40 osy (149.2 gsm).

TESTING BY HAND

Heating—Four specimens about 2 in. (5.1 cm) wide in the MD direction and about 140 mm long in the CD direction were cut from a larger piece of the laminate of this Example 2. Two marks 100 mm apart in the CD direction were placed on all samples. The samples were then heated in a hot-air circulating oven at 65.6°C for 1.5 min. The distance between the two marks on each specimen was then measured again and found to be, respectively, 56.0 mm, 56.0 mm, 53.0 mm and 57.0 mm, with an average of 55.5 mm. The samples were then stretched by hand until the tension in the samples indicated the specimens were fully extended, and the distance between the marks was again measured and found to be, respectively, 175.0 mm, 175.0 mm, 177.0 mm and 175.0 mm, with an average of 175.5 mm. The samples were then allowed to relax for two minutes and then again the distance between the respective pairs of marks was measured and found to be 90 mm, 94 mm, 89 mm and 96 mm, with an average of 92 mm.

The theoretical stretch based on necked spunbond by itself having about 82% stretch is 182/55.5=3.28 or 228% theoretical stretch. The actual stretch measured by hand was 175.5/55.5=3.16 or 216% actual stretch.

The difference in actual stretch of the extensible necked spunbond laminate of

Example 2 measured by hand, 216%, and the percent stretch in the extensible necked spunbond facing itself, 82%, was 163% (([216-82]/82) x 100=-163%), indicating substantially improved stretchability by using a heat shrinkable layer of elastic.

The difference in actual stretch between the extensible necked spunbond laminate of Example 2 and the conventional non-extensible spunbond laminate of Example 1 was 56.5% (([216-138]/138) x 100=-56.5%), indicating substantially improved stretchability by using the extensible necked spunbond.

TESTING BY MACHINE

20

A sample of the laminate measuring 18 inches (45.72 cm) in the MD and 22 inches (55.88 cm) in the CD was heat activated at 65.56°C for 3 minutes. After heat activation the sample measured 17 inches (43.2 cm) in the MD and 15.5 inches (39.4 cm) in the CD. The standardized WVTR of this heat activated material was 827. The theoretical CD stretch based on the necked spunbond by itself having about 82% stretch is 40/15.5 = 2.58 or 158%.

Five specimens were cut from the heat activated laminate. Each specimen was cut to 1 inch (2.54 cm) width in MD by 6 inch (15.24 cm) length in the CD, the latter length being in the direction of stretch measurement. Each specimen was tested by the Tensile and Stretch Test. The tensile strength in grams and percent stretch were measured for each specimen at various strain points on the curve, and the breaking tensile (break percent drop = 1%) and percent stretch at break (break drop elongation = 0.02 inches (0.05 cm)) were measured for each specimen. The averages of the tensile strength and percent stretch for the five specimens are shown in Table 2 below. The average percent stretch at break was 188%.

10

15

TABLE 2

										Break
% Stretch	0	25				125		175	184	188
Tensile Strength as (grams/2.54	0	539	759	895	1117	1485	1916	2210	2289	2261
cm)	_				_					

The difference in percent stretch at break of the extensible necked spunbond laminate and percent stretch in the extensible necked spunbond facing itself was 129% (([188-82]/82) x 100=-129%), indicating substantially improved stretchability by using a heat shrinkable layer of elastic.

The difference in average percent stretch at break between the extensible necked spunbond laminate of Example 2 and the normal non-extensible spunbond laminate of Example 1 (Comparative Example) was 55.4% (([188-121]/121) x 100=~55.4%), indicating substantially improved stretchability by using the extensible necked spunbond.

Although only a few exemplary embodiments of this invention have been described in detail above, those skilled in the art will readily appreciate that many modifications are possible in the exemplary embodiments without materially departing from the novel teachings and advantages of this invention. Accordingly, all such modifications are intended to be included within the scope of this invention as defined in the following claims.

It should further be noted that any patents, applications or publications referred to herein are incorporated by reference in their entirety.

CLAIMS

WHAT IS CLAIMED IS:

- 1. An elastic laminate material, comprising:
 - a) at least one first layer of an extensible material;
- b) at least one second layer of an elastic material;
 - c) said at least one second layer being laminated to at least a portion of said at least one first layer, said laminate being heat shrunk in the direction that is in the same axis as the direction that said at least one first layer is extensible.
 - 2. The elastic laminate material of Claim 1, wherein said elastic material is a film.
- The elastic laminate material of Claim 1, wherein said extensible material is a nonwoven material.
 - 4. The elastic laminate material of Claim 3, wherein said nonwoven material is a spunbond material.
- 5. The elastic laminate material of Claim 1, wherein said at least one first layer is necked in the cross direction.
 - 6. The elastic laminate material of Claim 1, wherein said at least one first layer is extensible in the cross direction and the machine direction.
 - The elastic laminate material of Claim 1, wherein said laminate is apertured at spaced apart locations.

8. The elastic laminate material of Claim 7, wherein aperturing is achieved by a method selected from the group consisting of pin aperturing, hydraulic needling, and ultrasonic bonding.

- 9. The elastic laminate material of Claim 1, wherein said at least one second layer is prestretched.
- 10. An elastic laminate material, comprising:

5

- a) at least one first layer of an extensible material;
- b) at least one second layer of an extensible material;
- c) at least one layer of an elastic material;
- d) said elastic layer being laminated to at least a portion of and between said first and said second layers, said laminate being heat shrunk in the direction that is in the same axis as the direction that the first and second layers are extensible.
- 11. The elastic laminate material of Claim 10, wherein said elastic laminate material has, in the same axis, stretch of from about 25% to about 350% more than said extensible material, as measured by hand testing.
 - 12. The elastic laminate material of Claim 10, wherein said elastic laminate material has, in the same axis, stretch of from about 50% to about 300% more than said extensible material, as measured by hand testing.

13. The elastic laminate material of Claim 10, wherein said elastic laminate material has, in the same axis, stretch of from about 100% to about 250% more than said extensible material, as measured by hand testing.

- 14. A breathable elastic laminate material, comprising:
- 5 a) at least one elastic first layer containing apertures; and,
 - b) at least one extensible substantially inelastic nonwoven second layer,
 said at least one elastic first layer being laminated to at least a portion of said at
 least one second layer, said laminate being heat shrunk in the direction that is in
 the same axis as the direction that said at least one second layer is extensible.
- 10 15. The breathable elastic laminate material of Claim 14, wherein said at least one first layer is at least one film layer.
 - 16. The breathable elastic laminate material of Claim 14, wherein said at least one first layer is a web selected from the group consisting of spunbond and meltblown materials.
- 17. The breathable elastic laminate material of Claim 14, wherein said at least one first layer is composed of a thermoplastic elastomer capable of being made latently shrinkable.
 - 18. The breathable elastic laminate material of Claim 14, wherein said at least one second layer is a web formed by a spunbond process.

19. The breathable elastic laminate material of Claim 14, wherein said at least one first layer comprises:

- a) a laminate of a thermoplastic elastomer capable of being made latently shrinkable, and
- 5 b) an adhesive.
 - 20. The breathable elastic laminate material of Claim 19, wherein said elastomer is shrunk by heating.
 - 21. The breathable elastic laminate material of Claim 19, wherein said adhesive is coextruded with said thermoplastic elastomer.
- The breathable elastic laminate material of Claim 14, wherein the basis weight of said breathable elastic laminate material is from about 0.4 osy (13.56 gsm) to about 6 osy (203.4 gsm).
 - 23. A breathable elastic laminate material, comprising:
 - a) at least one elastic first layer containing apertures; and
- b) at least two extensible substantially inelastic nonwoven second layers defined as a top layer and a bottom layer, said elastic layer being laminated to at least a portion of and between said top and said bottom layers, said laminate being heat shrunk in the direction that is in the same axis as the direction that the top and bottom layers are extensible.

24. The breathable elastic laminate material of Claim 23, wherein said breathable elastic laminate has a basis weight of from about 0.4 osy (13.56 gsm) to about 8 osy (271.2 gsm).

- 25. A process for forming an elastic material having high elasticity, comprising:
- 5 a) providing at least one first polymer,
 - b) forming said at least one first polymer into a first nonwoven web;
 - c) stretching said first nonwoven web to neck said first nonwoven web in the transverse direction;
 - d) providing a second polymer;
- 10 e) forming said second polymer into a second nonwoven web;
 - stretching said second web to neck said second nonwoven web in the transverse direction;
 - g) providing at least one layer of an elastic material that is heat shrinkable in the transverse direction;
- h) bonding said at least one layer of elastic material between said stretched first web and said stretched second web at selected locations to form a laminate; and,
 - i) heating said laminate of step h) so as to shrink said elastic material.
 - 26. The process of Claim 25, further comprising the step of forming apertures at least partially through said laminate.

INTERNATIONAL SEARCH REPORT

inton and Application No PCT/US 99/25972

A CLABBI IPC 7	PICATION OF SUBJECT MATTER B32827/12 D04H13/00		
According to		casion and IPC	
B. RELDS	SEARCHED		
iPC 7	countertation searched (closeification system followed by closeifica DO4H B32B	Ban ayribab)	
Documente	Son searched other than monthsum documentation to the extent that	such documents are included in the f	dide sparched
Bectronic d	nta base consulted during the international seconds (name of data);	see end, where precioel, search term	e used)
C. DOCUM	ENTS CONSIDERED TO BE RELEVANT		
Category *	Citation of document, with indication, where appropriate, of the r	olovent passages	Relevant to clubs No.
X	DE 195 23 497 A (CORONOR COMPOS) 2 January 1997 (1997-01-02) column 2, line 63 -column 3, linexamples	•	1-4,6, 9-13,25
Υ .	claims		1,7,8, 14-18, 23-26
X	EP 0 217 032 A (KIMBERLY CLARK 0 8 April 1987 (1987-04-08) claims 31-55 figure 2A	(O)	1-4,6, 9-13,25
Υ -	DE 42 43 012 A (COROVIN GHBH) 23 June 1994 (1994-06-23)		1,7,8, 14-18, 23-26
	column 6, line 47 - line 50 claims		
		<u>-/-</u>	
X Fun	har documents are Bated in the combrustion of box C.	Y Petent femily members ass	Basted in corners.
conside	degories of cited documents : Est dollaring the general dutie of the est which is not beed to be of particular relevance document but published on or either the international	"I later document published after to or priority date and not in confi- cited to understand the principal investigan	ict with the application but e or theory underlying the
filing d "L" docume which		"X" document of particular refeveno carrot be considered novel or brothe an inventive step when "Y" document of particular refeveno carrot be considered to involve	current be considered to the document to taken alons at the cisimed invention
others The decume	ent referring to an onal disclosure, usa, entitation or means ent published prior to the International Ring date but	document le combined with on pertie, such combined on being in the ext.	e or more other: such docu- j obvious to a person diffed
	han the priority date claimed ectual completion of the International creach	"&" document member of the came Date of melling of the internation	
2	9 February 2000	07/03/2000	
Name and o	meting address of the ISA European Paters Office, P.B. 6818 Patersisen 2	Authorized officer	·
	HL - 2280 HV Ribselft Tel. (+31-70) 340-2040, Til. 31 651 epo nil, Fizi: (+31-70) 340-3016	De Jonge, S	

27. The process of Claim 25, wherein said heating step is done at about 150°F (65.5°C) for about 1.5 minutes.

INTERNATIONAL SEARCH REPORT

betos. Incl Application No PCT/US 99/25972

C.(Continuation) DOCUMENTS CONTINUENTO BE RELEVANT								
Calegory *	Otation of document, with Indication, where appropriate, of the relevant passages		Fictoverst to delth No.					
A	US 4 446 189 A (ROMANEK GERALD A) 1 Hay 1984 (1984-05-01) claim 1		1,10,14, 23,25					
A	EP 0 788 873 A (COROVIN GHBH) 13 August 1997 (1997-08-13) claims 1,2		1,10,14, 23,25					
			-					
·			·					
		-						
		ż						
		. •						
,		·						

Q. 6. 0

INTERNATIONAL SEARCH REPORT

information on patent family recrebers

Inton and Application No PCT/US 99/25972

Patent document cited in search repor	ı	Publication date	l	Patent family member(s)	Publication date
DE 19523497	A	02-01-1997	HOM		
EP 0217032	A	08-04-1987	US	4720415 A	19-01-1988
			AU	591507 B	07-12-1989
			AU	6048686 A	05-02-1987
			CA	1261723 A	26-09-1989
			DE	3683924 A	26-03-1992
			JP	1956246 C	28-07-1995
			JP	6086712 B	02-11-1994
			JP	62033889 A	13-02-1987
			KR	9308255 B	27-08-1993
			нX	167432 B	22-03-1993
DE 4243012	A	23-06-1994	AT	138850 T	15-06-1996
			AU	56 92294 A	19-07-1994
			BR	9307673 A	08-09-1999
			CA	2150366 A,C	07-07-1994
			CZ	9501395 A	13-12-1995
			HO.	9414607 A	07-07-1994
		•	DE ·	59302850 D	11-07-1996
•			DK	674581 T	07-10-1996
			EP	0674581 A	04-10-1995
			ES	2089908 T	01-10-1996
			FI	952983 A	16-06-1995
•			GR	3020954 T	31-12-1996
			HU	72562 A	28-05-1996
			JP ·	8504693 T	21-05-1996
			KO.	952421 A	16-06-1995
			US.	5683787 A	04-11-1997
US 4446189	A	01-05-1984	ІКОИ	.	
EP 0788873	A	13-08-1997	DE	19604956 A	14-08-1997
		•	JP	9327887 A	22-12-1997
			US	5939178 A	17-08 -199 9

Form PCT/IBA-010 (posture torridy crystal) (July 1980)